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DMIC

ALLOY DEVELOPMENT

Air Force Wheel/Bucket Alloy Development

Universal-Cyclops has accomplished the initial production scaleup of Unitemp AF 2-1DA by melting three 3000-pound heats.(1) However, because of the tight chemical analysis range and lack of standards for this new alloy, only the third heat was within the specified chemi-try. The chemical range, aim, and analysis for the third heat are given below.

Preliminary hot-working experience was gained on one of the off-chemistry heats. The working-temperature range was 2050 to 1850 F.

Air Force Hot Compressor Alloy Development

The objective of this General Electric program is to develop ultrahigh-strength alloys for use as a disk alloy in the aft stages of the compressor in advanced turbojet engines.(2) Problems encountered in hot rolling or forging of the five alloys selected initially and screened for tensile and creep-rupture properties and microstructural stability, were solved by cladding during working. One alloy, No. 3, has been selected for thermomechanical processing studies. The composition of Alloy No. 3 is as follows: 0.15 C, 3.5 Al, 2.5 Ti, 15 Cr, 8 Co, 3.5 Cb, 3.5 W, 3.5 Mo, 0.05 Zr, 0.01 B, balance Ni. The thermomechanical processing study was considered necessary to determine the optimun processing procedure, since the ductilities of the alloys were lower than desired for compressor disk application.

AF 2-1DA and Rene' 85 have been added to this program and are being processed for study. Subsequently, the three best alloys will he further evaluated, particularly for creep and interrupted, low-cycle fatigue. The best alloy will then be more extensively evaluated, including evaluations in 1000 hour creep and stress rupture tests.

Sulfidation Resistant Alloy

Preliminary information on a new experimental International Nickel alloy, IN-738X, indicates that it has minimum mechanical properties equal to those of Alloy 713C, combined with greatly improved corrosion resistance and freedom from embrittling microstructural phases.(3) This alloy is not yet in commercial production although two large heats have been produced to supply material for commercial evaluation. Its nominal chemical composition is as follows:

Element	Weight Percent	Element	Weight Percent
C	0.17	Cb	0.90
Co	8.50	Al	3.40
Cr	16.00	Ti	3.40
Mo	1.75	B	0.01
W	2.60	Zr	0.10
Ta	1.75	Ni	Balance

Iron, silicon, manganese, and sulfur should be as low as possible. The melting range for this alloy is 2250 to 2400 F, and the recommended heat treatment is 2050 F for 2 hours, air cooled, plus 1550 F for 24 hours followed by air cooling.

Figure 1 shows a Larson-Miller parameter plot for IN-738X, IN-100, and Alloy 713 C. No sigma-phase was observed, optically or by X-ray diffraction, in a stress-rupture test specimen after 3946 hours at 1500 F and at a stress of 40,000 psi.

	C	P	S	Si	Mn	Zr	B	Fe	Al	Ti	Cr	W	Mo	Co	Ta	Ni
Range	0.33 0.37	0.015 max.	0.015 max.	0.10 max.	0.10 max.	0.05 0.15	0.012 0.017	0.50 max.	4.50 4.70	2.80 3.20	11.5 12.5	5.80 6.20	2.80 3.20	9.50 10.50	1.30 1.70	Balance
Aim	0.35	—	—	—	—	0.10	0.015	—	4.60	3.00	12.00	6.00	3.00	10.00	1.50	Balance
Heat K67490	0.34	0.001	0.008	0.01	0.01	0.52	0.016	0.18	4.66	3.15	11.73	6.12	2.88	9.98	1.64	Balance

Compositions of Alloys Released

AiResearch has just released the compositions of its tantalum- and yttrium-containing alloys AiResist 213 and AiResist 215.(4)

Alloy	C	Mn	Cr	Ni	Co	W	Ta	Al	Fe	Y	Zr
AiResist 213	0.2	0.1[a]	20	0.5[a]	Bal.	4.5	6.5	3.5	0.5[a]	0.10	—
AiResist 215[b]	0.35	0.2[a]	19	0.5[a]	Bal.	4.5	7.5	4.3	0.5[a]	0.10	0.10

(a) Maximum (b) Cast alloy

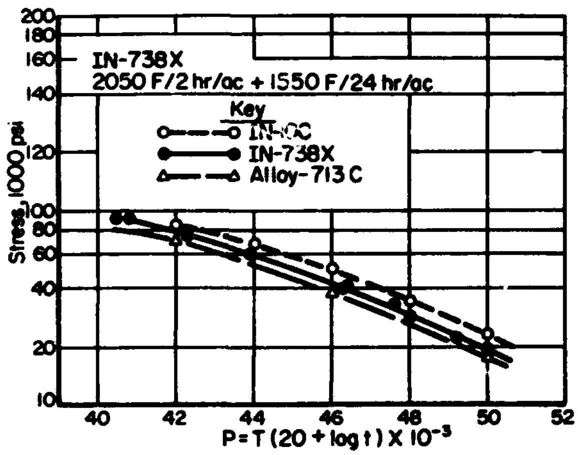

FIGURE 1. LARSON-MILLER PARAMETER CURVES COMPARING IN-100, IN-738X, AND ALLOY-713C(3)

Rupture strengths have been available for some time. They are as follows:

Rupture Strength, 1000 psi

	1200 F		1500 F		1800 F		2000 F	
	100 hr.	1000 hr.	100 hr.	1000 hr.	100 hr.	1000 hr.	100 hr.	1000 hr.
AiResist 213	65	—	20	13	5.0	3.5	2.8	—
AiResist 215	—	—	22	15	8.0	6.0	4.0	3.4

AiResist 213 sheet and tubing is intended for combustion-chamber liners and heat-exchanger parts. AiResist 215 is a casting alloy intended for gas turbine nozzle guide vanes. Both alloys are resistant to hot corrosion.

ALLOY STABILITY

Microstructural Instability of Nickel-Base Alloys

In a study of instability of nickel-base superalloys, TRW determined whether stress promotes or retards the formation of phases such as sigma and mu.(5) Five alloys were included in this study, IN-100, B-1900, U-700, René 41, and Unitemp AF2-1D. Stress appeared to have an effect on the microstructural stability of only two of the five alloys studied. These alloys were IN-100 and U-700, both sigma formers. These results indicated that there is an optimum stress that will promote sigma formation and varies with temperature, and that a sufficiently high stress will retard sigma formation. Further studies are being contemplated to verify these results using a heat of U-700 with a greater sigma forming potential. Mu phase, which formed in René 41 and Unitemp AF 2-1D, did not appear to be affected by stress.

Oxidation of Nickel-Base Superalloys

General Electric is conducting an investigation to thoroughly identify and demonstrate an approach for improving the surface stability of turbine-blade materials through minor additions of Group III B Metals, rare earth metals, thorium, and manganese.(6) The alloy bases selected for evaluation are (1) a cast alloy René 100 (IN-100 with composition modified for maximum internal stability) and (2) a development wrought alloy, Unitemp AF 2-1DA. These alloys have been selected as the basic alloys for study since they are among the strongest available cast and wrought alloys with high potential for use as turbine-blade and wheel materials in advanced engines. These alloys are also representative of the new generation of nickel-base alloys currently being developed.

Six rare-earth elements, each at two concentration levels, are being added to René 100, and five rare-earth elements, also at two concentration levels, are being added to AF 2-1DA alloy. Considerable difficulties were encountered during the melting and processing of the doped alloys and some remelting will be necessary. Analysis of doped René 100 alloys indicated the rare earths to be present as a Ni_5(R.E.) phase. The oxidation behavior of the base Unitemp AF 2-1DA alloy has been determined and has been found to be comparable with other commercial nickel-base alloys. Preliminary cyclic oxidation results of doped René 100 alloys show improved scale adherence at 1800 F.

PROCESS DEVELOPMENT

Manufacturing Process Development for Superalloy Cast Parts

Abex has a continuing Air Force project to develop methods for the manufacture of large superalloy castings with reproducibly high integrity and with dimensional tolerances within the category of "precision" castings.(7) Current effort is directed mainly at Alloy 713LC, with some additional effort on Alloy 718 and René 41.

An investigation of the microstructure of Alloy 713LC, Alloy 718, and René 41 supports the conclusion that Alloy 713LC will be the alloy most adaptable to the wide range of cooling rates and section sizes that will be encountered in the manufacture of large superalloy castings.

Heating to 1200 F for up to 100 hours has no detrimental effect on the properties of cast Alloy 713LC, and has produced a significant increase in yield strength with no other changes of significance.

The room-temperature and 1000 F low-cycle fatigue characteristics of Alloy 713LC have been established and appear to compare favorably with other materials currently in use as turbine-rotor alloys.

Process Development for Improved High-Strength Superalloy Sheet

The Union Carbide program to develop a process for the production of high-strength Udimet 700 alloy sheet has continued.[8] Three melting and casting techniques, vacuum induction melting (VIM), consumable electrode vacuum remelting (CEVM), and electroslag remelting (ESR), were used in Phase I for the production of ingots and slabs. These were, in turn, reduced to sheets 0.030 inch, 0.020 inch, and 0.015 inch thick by 22 inches wide. Sheets of 0.030-inch and 0.020-inch gage, three feet wide and 6 feet long will be produced during Phase II.

Homogenization tests on ESR specimens showed the temperature for complete solutioning of all carbides and gamma prime to be 2150 to 2200 F.

The tensile properties from tests conducted in argon at 1400 and 1600 F were found to be superior to those in air, while the differences in properties tended to decrease as the thickness of the sheet material decreased.

The Udimet 700 alloy sheet was observed to be thermally stable at exposures of 100 hours at temperatures up to 1600 F. Loss in properties after 100-hours' exposure in air at 1800 F are explained by a depletion of minor elements at the surfaces of the material, and overaged precipitates in the matrix and at the grain boundaries.

Development of a Process for Electroslag Melting and Casting of Materials

The purpose of a melting project at Mellon is to develop a manufacturing process for the electroslag consumable-electrode remelting and casting of 18 percent nickel maraging steel, 300 ksi grade ultrahigh strength steel, René 41, Alloy 713C, and Udimet 700 in the form of round and slab ingots for conversion to flat-rolled products.[9]

The ability to conduct a reproducible melting procedure has been demonstrated. The electroslag melted material has displayed a high level of hot workability. Mechanical-property evaluations of the hot-worked-plate products from 7-inch-diameter ingots of maraging steel and René 41 have been completed. Also, a second 7-inch-diameter ingot of René 41 has been electroslag remelted, and its excellent hot workability has been demonstrated by forming a section of this ingot into a 21-inch-diameter ring.

Scaled-up remelting of Udimet 700 to a 7-inch-diameter ingot has been accomplished. This ingot was successfully hammer forged to an intermediate 3-inch by 6-inch slab. It will be further reduced by direct hot rolling to a 1-inch-thick plate.

Two 1500-pound slab ingots of René 41 have been electroslag melted. A section of the first ingot has been hot rolled to 1-inch-thick plate, while the second ingot has been press forged to a 7-inch by 11-inch billet. This intermediate shape is being held until the plate product from the first ingot has been evaluated.

Superalloy Strip by the Slip Casting Process

The Chrysler program to develop the continuous production of metal strip and sheet by the slip casting process will make use of four superalloys, U-700, IN-100, X-40, and Waspaloy.[10] At present, the study is concerned with the optimization of metal slip compositions, casting and drying, sintering and rolling, and density. Mechanical properties of superalloy strip produced by this method have not yet been determined.

DISPERSION-STRENGTHENED ALLOYS

Dispersion-Strengthened Metal Structural Development

Douglas Aircraft is studying the possibility of using dispersion-strengthened (DS) nickel-and cobalt-base alloys to replace coated refractory-metal components for short-lived structural applications in the 2000 to 2400 F temperature range.[11] Figure 2 shows a comparison of 100-hour rupture strengths of Ni-20Cr-2ThO$_2$, Hastelloy X, Haynes 25, René 41, and coated Cb-752. This comparison shows the advantage of TD NiC over the superalloys at temperatures over 1600 F. The 100-hour rupture

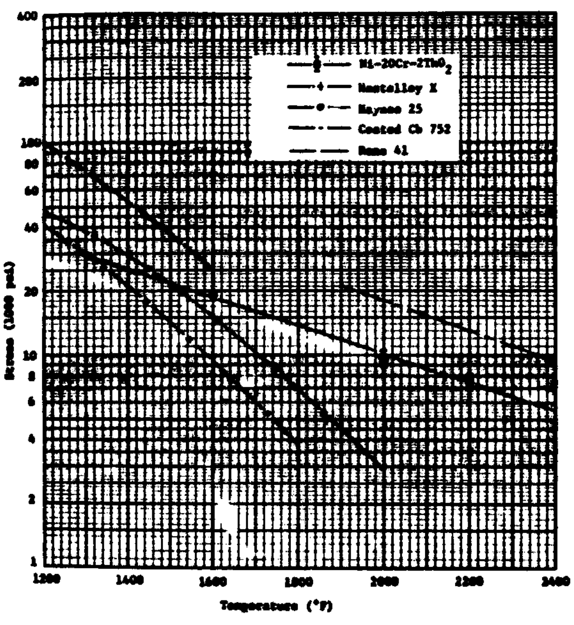

FIGURE 2. COMPARISON OF 100-hr. STRESS RUPTURE PROPERTIES FOR TD NiC, COATED Cb-752, AND SUPERALLOYS[11]

stress for TD NiC, however, is only a little over half that of Cb-752. Joining and fabrication tests are being conducted on TD NiC structural configurations of honeycomb-sandwich, single-face, corrugation-stiffened panels, and a subscale hot-gas duct segment.

Development of a Manufacturing Process for Thin Sheet and Foil

Du Pont has developed a foil process comprising warm-pack rolling of 0.020-inch TD Nickel-Chromium sheet to an intermediate gage and cold tension rolling to final gage.(12) This process which produced 12-inch-wide foil is now being scaled up to about 24 inches wide. Foil of 0.005-inch thickness of excellent quality has been produced. In warm pack rolling, a wipe coating of colloidal silica was found to be effective as a parting agent. Milk of magnesia is difficult to remove and should not be used.

Sheet breakage in tension rolling has been a major problem, and satisfactory rolling required surface sanding of some intermediate-gage sheets and edge conditioning of all sheets. Frequent in-process anneals in the 1300 to 2200 F range produce the best high-temperature properties in cold-rolled foil. The optimim cold rolling-annealing schedule for best foil properties has yet to be determined.

Development of a Dispersion Strengthened Nickel-Base Alloy Using the High Intensity Arc Process

Alloy powders of nominal composition 60 Ni, 20 Co, 10 Mo, 10 W and containing dispersions of thoria, yttria, alumina, magnesia, and lanthana were prepared and evaluated by Vitro Laboratories.(13) Based on their dispersion characteristics, thoria and yttria dispersions were selected for larger scale preparation. Billets containing 0, 2, 4, and 7 volume percent thoria and yttria were prepared and evaluated by density and microhardness measurements as well as optical and electron metallography before and after thermal-stability tests. The best of the billets produced contained 4 percent thoria. The yttria-containing billets showed particle growth and inhomogeneous distribution. Clad billets without a dispersion oxide (control material) and containing 4 percent thoria were extruded to bar for stress-rupture testing.

This material had a 100-hour rupture strength less than one-tenth that of TD Nickel and little better than that of the control material. The reason for the poor rupture strength was not determined.

EVALUATION AND APPLICATIONS

Superalloys for Honeycomb Heat Shields

Solar is evaluating foil-gage superalloys René 41, Alloy 718, Inconel 625, Haynes Alloy No. 25, TD Nickel, and TD NiC as candidate materials for structural and heat-shield panels for re-entry and hypersonic vehicles.(14) Operative degradation mechanisms of the alloys are to be studied so that reuse capabilities and limitations can be predicted. Evaluation will involve characterization of alloy degradation mechanisms after exposure to slow thermal cycles in dry air, atmosphric pressure air, and in air at reduced pressures of 10 and 0.18 torr, the equivalent pressures at altitudes of 100,000 and 200,000 feet, respectively.

Baseline-data tests completed are tensile tests up to 2000 F, notched-tensile and fatigue tests at room temperature,and creep tests up to 2400 F. At 2000 F, higest and lowest yield strengths were exhibited by TD NiC (16.1 ksi), and René 41 (4.3 ksi), respectively, and the highest elongation by Haynes 25, 5.7 percent. Axial tension-tension fatigue tests at room temperature on foil-gage specimens gave the following approximate fatigue limits (5×10^6 cycles):

Superalloys	Fatigue Limit, (5×10^6 cycles)
Rene' 41	82
Alloy 718	75
Inconel 625	82
Haynes No. 25	75
TD Ni	48
TD NiC	65

Creep tests in air atmosphere at 1800 to 2400 F to determine the stress necessary to produce 1 and 5 percent creep in 30 hours or less have been completed. From the point of view of this program, the recommended maximum service temperatures, determined from creep-rate considerations, are:

2000 F for Alloy 718 and René 41
2100 F for Inconel 625 and Haynes 25
2400 F for TD Ni and TD NiC

With the possible exception of TD NiC, these temperature limits are 100 to 200 F above the usually recommended temperature limits for these alloys. Therefore, they should be applied only to low-stress short-time applications.

Alloy 718 for Bellows and Gimbal Applications

A study was made by Solar to investigate the effects of thermal treatments on Alloy 718 and its subsequent fabrication processes.(15) Tests have shown that there is considerable difference in heat-affected-zone cracking behavior from heat to heat and in heats from various producers. The increase in cracking susceptibility of one as-received material over another has been attributed to increased amounts of grain boundary segregation, particularly in areas of titanium-columbium carbo-nitride stringer formations. Microcracking in the heat affected zone followed the general pattern of the stringer formations.

The thermal treatment that resulted in the least heat-affected-zone cracking in the two thicknesses evaluated (0.040 and 0.209 inch) was the Pratt & Whitney-developed thermal treatment (AMS-5596). This cycle consists of annealing at the relatively low temperature of 1750 F in a suitable protective atmosphere followed by double aging treatment at 1325 and 1150 F.

NEW TECHNICAL NOTE

The following technical note giving the currently available information and data on TD Nickel-Chromium is now available from the Defense Metals

Information Center:

TD Nickel-Chromium, November 2, 1967.

REFERENCES

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(10) Preliminary information reported by Chrysler Corporation, Detroit, Mich., under U. S. Air Force Contract AF 33(615)-3998.

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(12) Preliminary information reported by E.I. du Pont de Nemours & Company, Baltimore, Md., under U.S. Air Force Contract F 33(615)-67-C-1015.

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(15) Valdez, P.J.,"Evaluation of Thermal Treatments for Nickel-Base Inconel 718 Alloy in Bellows and Gimbal Application", Final Report RDR 1460-1, Solar Division, International Harvester Company, San Diego, Calif., Contract NAS 8-11282 (July 1967) DMIC No. 69938.

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R. W. Endebrock, Editor